Composition Analysis

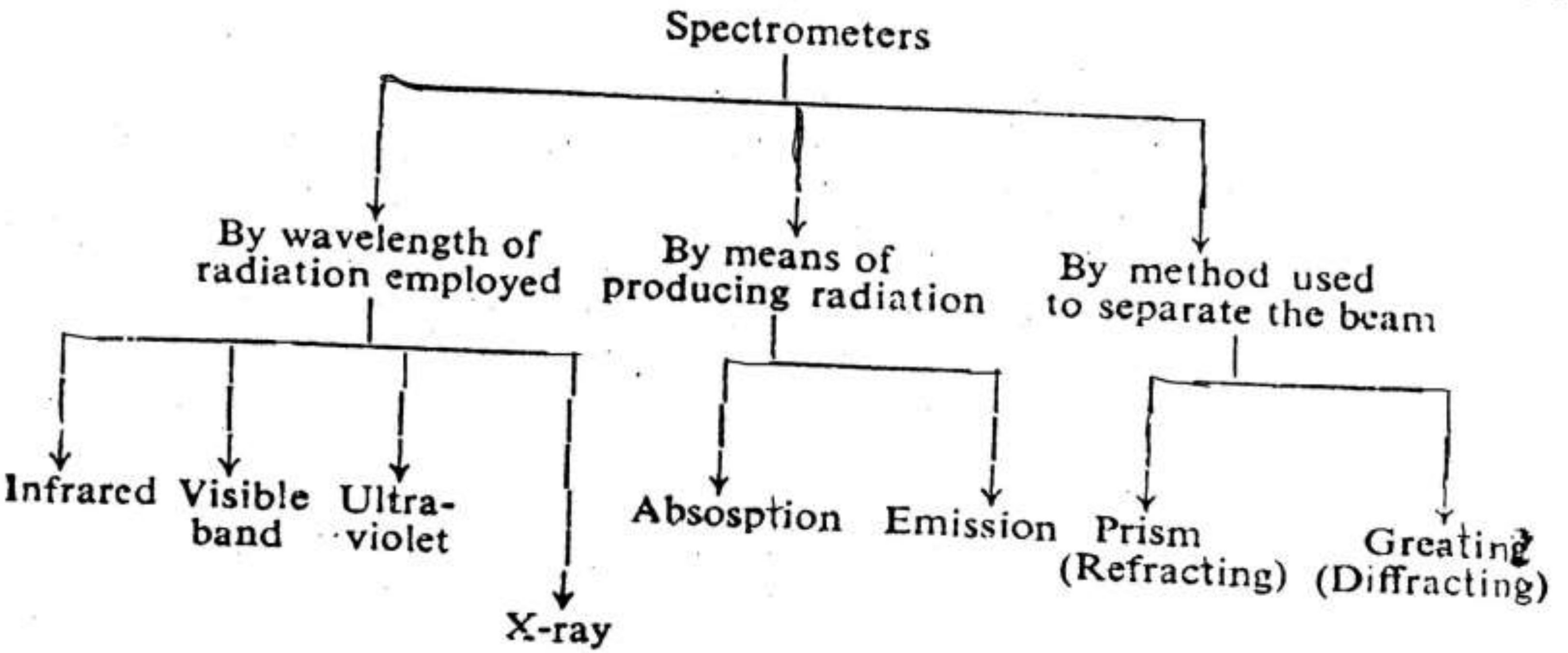
Methods of Composition Analysis

- Spectroscopy- for solids, liquids and gases
- X-Ray Diffraction- for solids
- Thermal conductivity – for gases
- Polarography – for fluids
- Chromatography – for fluids

Spectroscopy

- Fundamental properties of a substance are identified by the atomic or molecular spectra.
- Atoms of a substance are excited, when they either absorb or radiate energy at unique frequencies. The electrons absorbs or radiate discrete amount of energy and shift to a new configuration. These amount of radiation is measured and used for identifying the substance as the band gap between different spectrum is different for each material.

Spectrometers

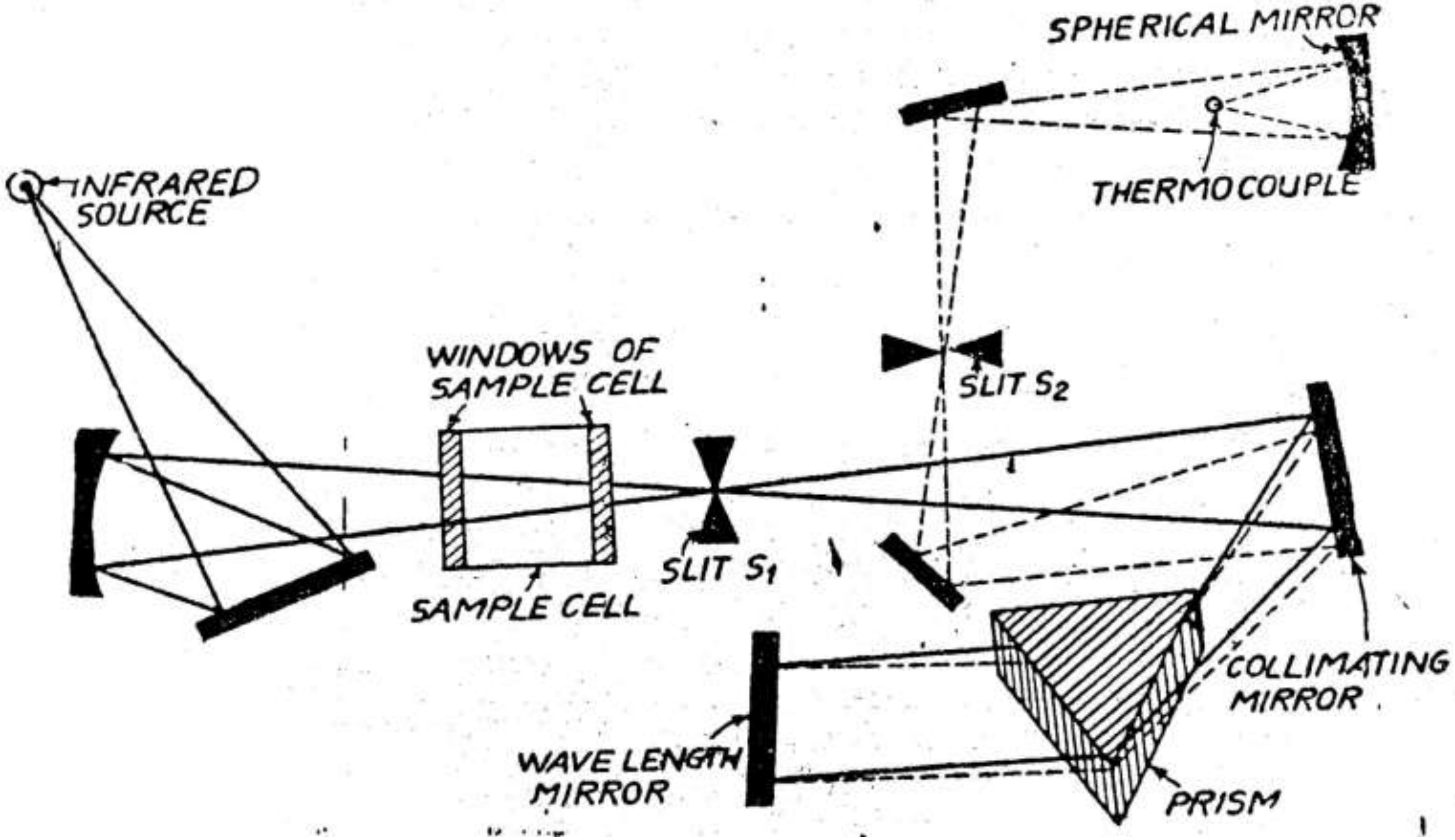


Absorption spectroscopy

- when radiation is passed through a sample of unknown substance, certain frequencies are absorbed. Absorption lines are dark lines on a light background on a photographic negative.
- According to type of radiation – three types

1. Infra red – 1 to 25 microns wavelength
2. Ultraviolet – 0.2 to 0.8 microns wavelength
3. X ray – 0.4 to 1.5 Armstrong wavelength

Infra red spectroscopy

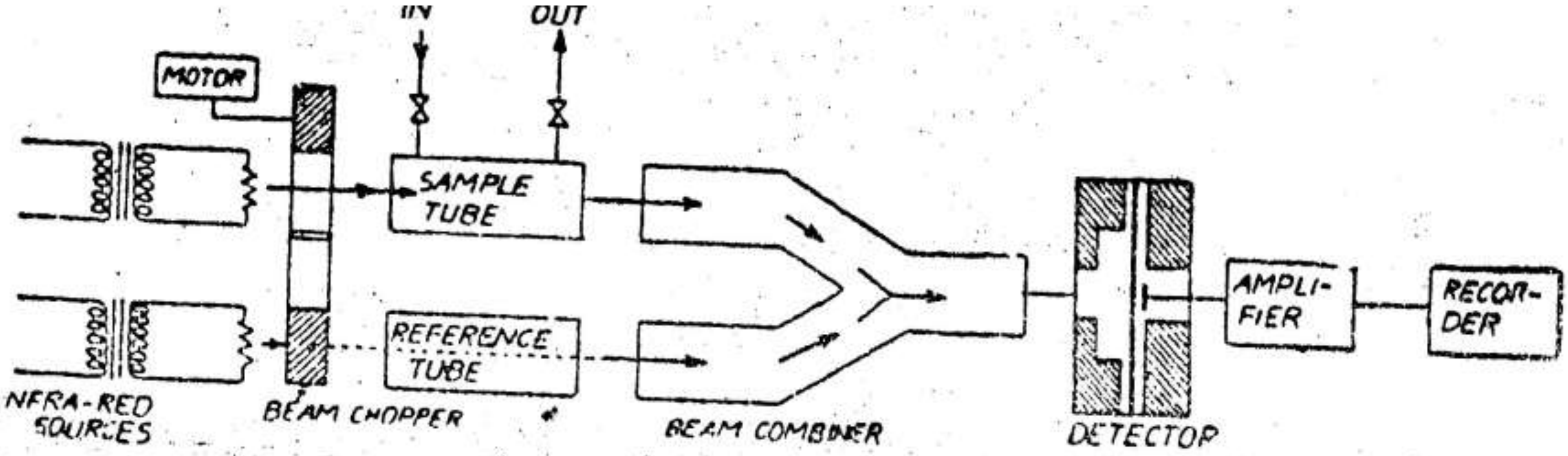


Operation

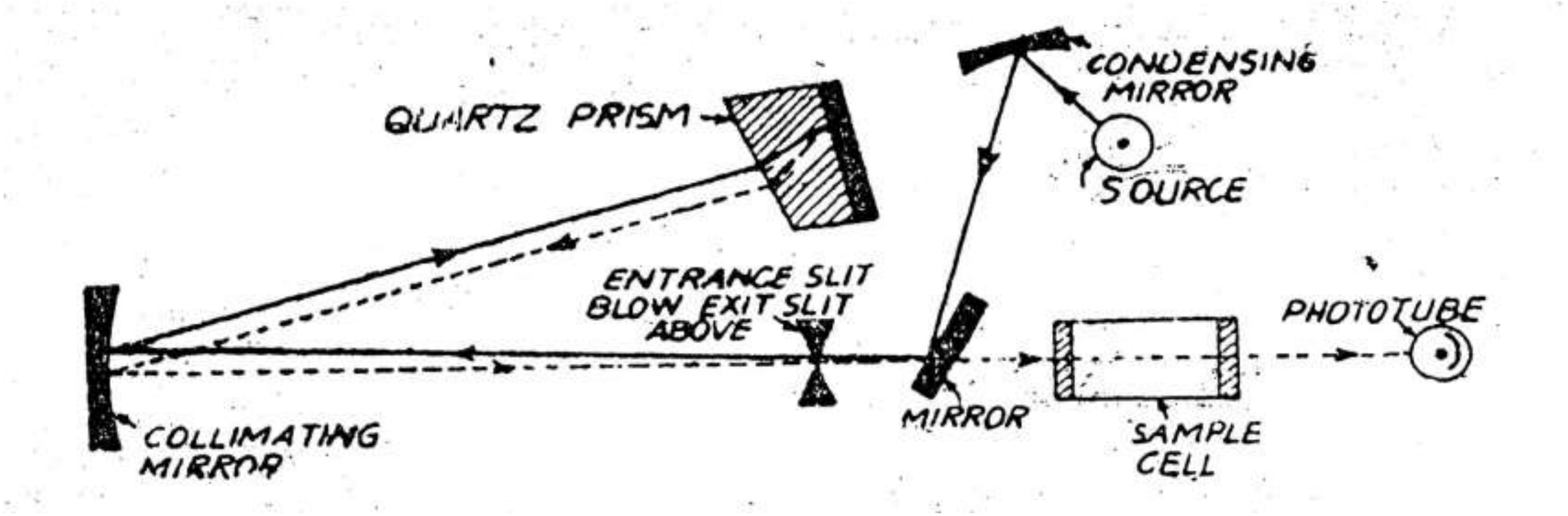
- Infra red radiation is provided by an electrically heated resistor. The beam passes to a mirror, then into a parabolic mirror, and after passing through the sample cell, falls on slit 1. The beam then passes to a collimating mirror, where it is rendered parallel, and then through a prism, where it is refracted. The wavelength mirror reflects the beam back through the prism, where it is once more refracted. The beam is next reflected by a collimating mirror to a plane mirror and then to slit2. The beam is finally reflected to the thermocouple, where the beam intensity is measured.
- By slowly rotating the wavelength mirror, entire wavelength spectrum can be scanned. The thermocouple will measure the intensity of beam at each wavelength.

- Source – black body at 1000°C
- Prisms – Lithium flouride – 1 to 5.5 u, Calcium flouride – 2 to 8.5 u, Sodium flouride 3 to 15 u , Potassium bromide – 15 to 25 u.
- Application- almost all substances especially petroleum products, rubber, hydrocarbons, pharmaceuticals, inorganic and organic chemicals can be analysed.

Schematic diagram



Ultra Violet spectroscopy



Operation

- An image of light source is focused by the condensing mirror and by flat mirror on the entrance slit. The portion of the spectrum passing through the exit slit then passes through the sample cell and falls on a phototube. The intensity of the beam is measured by a suitable amplifier connected to an automatic balance potentiometer instrument.
- Source- tungsten filament (visible region 0.3 to 1 u) or hydrogen discharge lamp for uv region 0.22 to 0.35u)
- Prism- Quartz
- Applications – vitamin compounds, benzene and toluene in petroleum distillates, butadiene, hydrocarbons, ethylene, carbonyl compounds, poly saturates, fatty acids

X ray absorption spectrometer

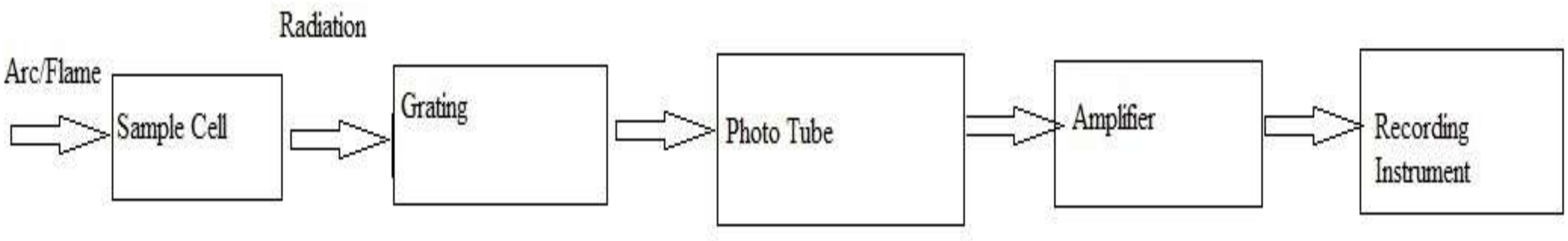
- X ray source is used in the analysis of elemental components of metals and alloys. Sulfur content of oil, lead in gasoline, plastics analysis, filler content of rubber and wood.
- Source X-ray tube providing radiation in 0.4 to 1. 5A
- Two beams are taken from the source. One passes to a sample cell to a fluorescent screen. The other beam passes through a variable thickness Al attenuator and then through a second sample cell to the same fluorescent screen. An amplifier is used to measure the difference in intensity of two beams and Al attenuator is adjusted to have same intensity for 2 beams. The thickness of Al can be calibrated to the content of critical component

Emission spectroscopy

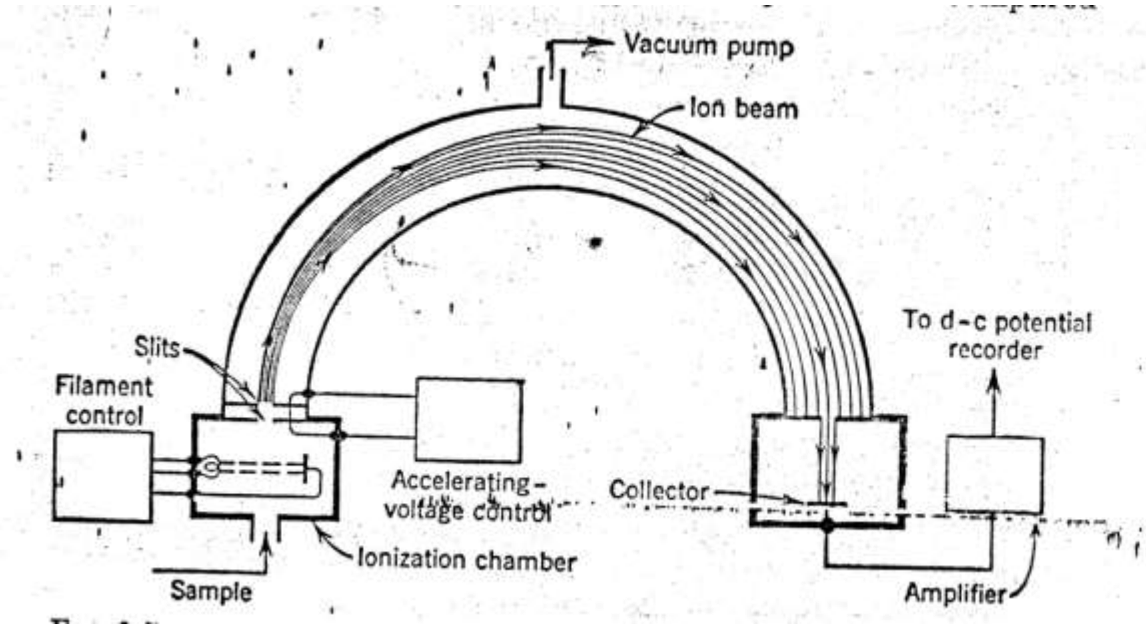
- An unknown substance is placed in a flame or arc. This causes emission of radiation which is characteristic of each substance. By collecting the emitted radiation and separating by refraction or diffraction, the intensity of radiation at each frequency is measured. Emission lines are bright lines on a dark background on a photographic negative.
- two types

1. Arc type – for mg, al and steel- an arc sperk is used to emit the radiation and it is diffracted using a grating (800-2500A)
2. Flame type – inorganic compounds, Boron, Ca, Ce, Na, K, Co, Li,Fe, Mg, Mn, Ni - aqueous solution is atomised and introduced into a burner. A lens system concentrates the emitted light through filters on a phototube.
3. Fluorescence- pharmaceuticals, vitamin compounds where photo deterioration is to be avoided

Schematic Diagram



Mass spectrometer



Principle

- A sample of unknown substance (vapor or gas) is bombarded by an electron beam in an evacuated chamber. The bombardment produces ions by knocking off an electron from each of the various kinds of atoms present in the substance. These ions are then accelerated into a circular path, using a magnetic field, the radius of which varies with the mass of ions and thus ions are sorted into beams. By measuring the strength of each beam, all the various kinds of atoms can be counted and identified.

Applications

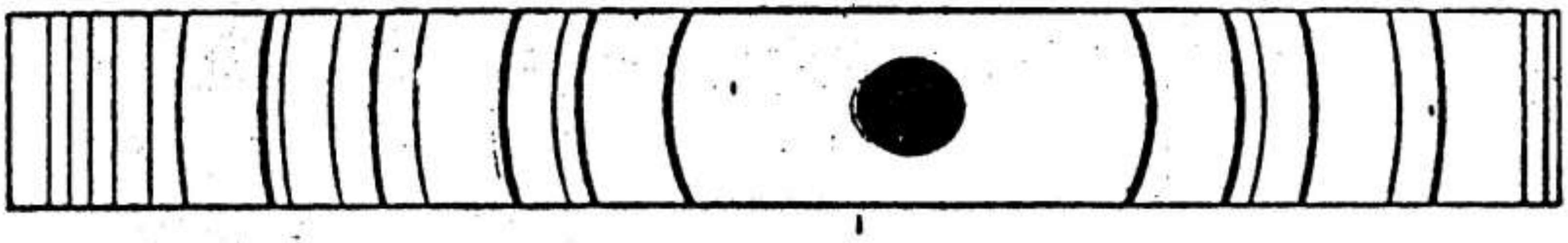
- Analysis of aliphatic and cyclic hydrocarbon compounds
- Synthetic rubber manufacture
- Petroleum refining
- Analysis of light gases, olefins, propylene
- Liquid and gaseous pharmaceuticals
- Chemical Manufacturing

X- Ray Diffraction

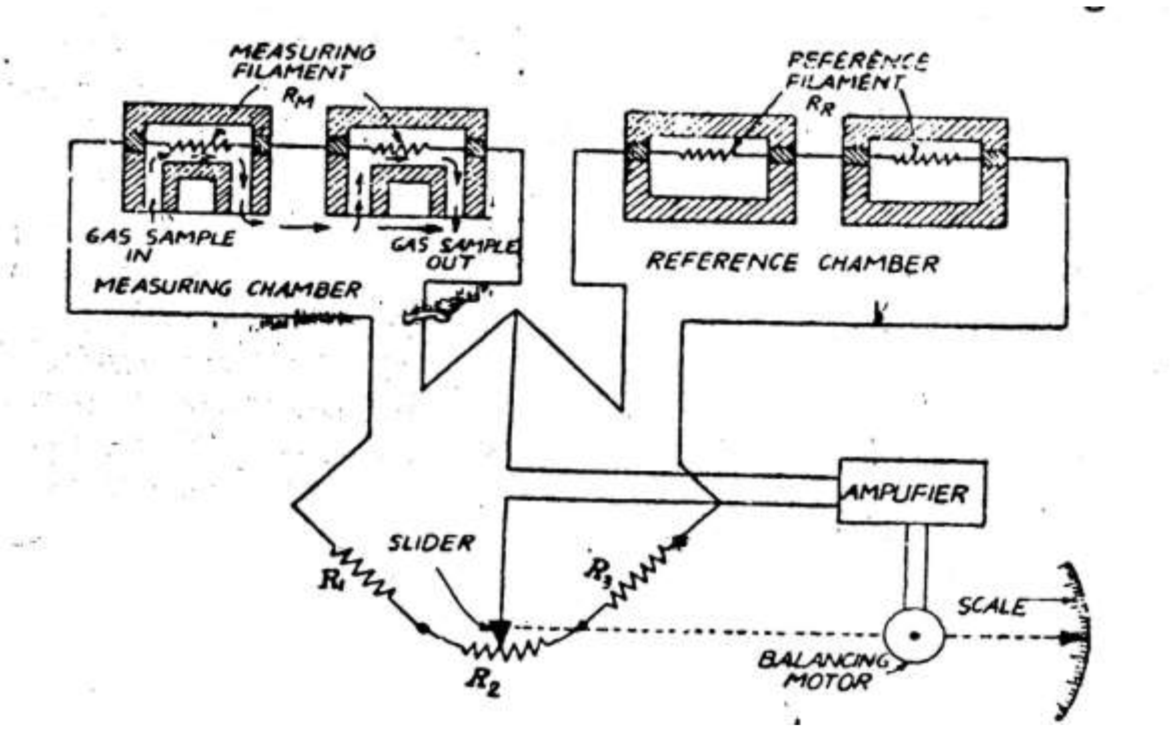
- In many crystalline and amorphous solids, the atoms of each element lie in regular spacings called space lattices.
- If X-ray of one frequency (monochromatic) is directed to a sample of solid at a specified angle, the radiation emerges from the solid in several beams forming a pattern.
- This is bcoz some of the radiation is reflected by certain planes of atoms & some is reflected in another direction by other planes.
- Therefore a photograph of diffracted beams will indicate certain arrangements and spacings of atoms of the substances
- XRD pattern is unique for any solid bcoz, the arrangement and spacing of the atom in the molecule in the crystal are different for different solids

- Applications- control of dyes & fillers in rubbers, selection of Mn ores, analysis of compounds and particle size for research purposes

XRD Pattern

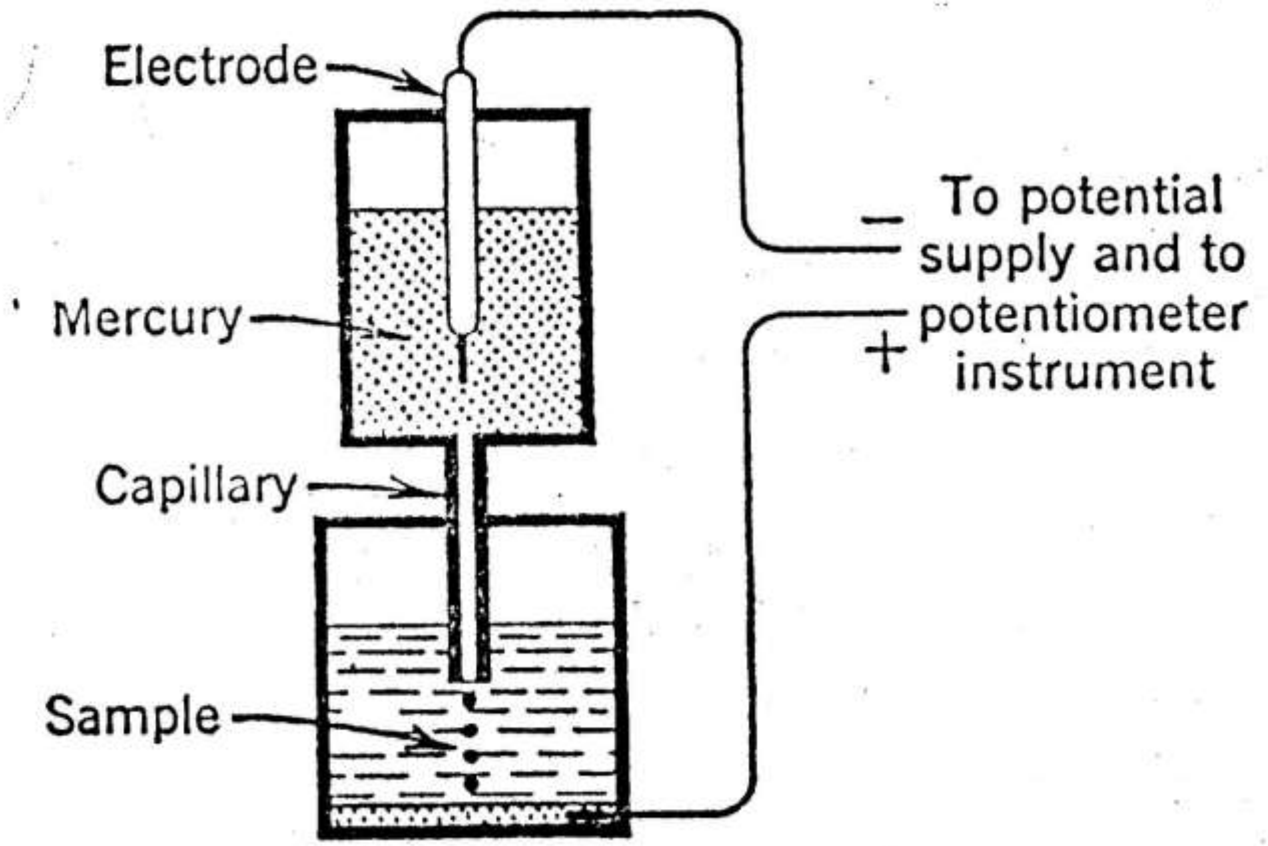


Thermal conductivity gas analyzer



- Gases differ the ability to conduct heat (k of air – 1.0, CO_2 – 0.585, C0 – 0.958, He – 6.08, H_2 – 7.35 etc.)
- Thermal conductivity cell consists of four chambers. The two chambers for measuring cell and other two for reference cell In measuring cell the sample gas is made to flow and the other cell is sealed and contains a reference gas such as air.
- The temperature of the filament in the measuring cell rises as the thermal conductivity of the gases passing around it decreases. Both the cells are connected to a Wheatstone bridge as shown in figure.

Polarograph

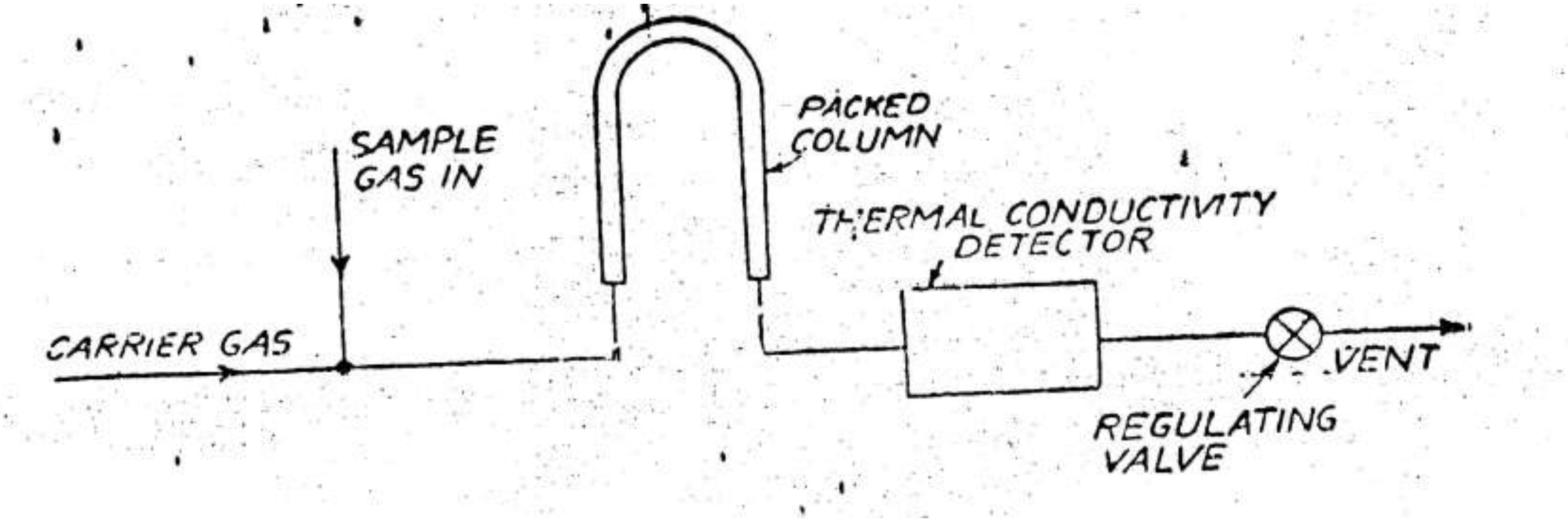


Principle- electrolysis

- It consists of an electrolytic cell with two Hg electrodes.
- Anode-Still electrode- a pool of Hg at the bottom of the cell
- Cathode- dropping electrode- Hg is dropping from the capillary tube & hence the surface is constantly renewed.
- When a gradually increasing potential is applied to a mixture of metallic ions, only a small current will flow through the cell initially. When the potential reaches the discharge potential of one of the ions, the current flow will rise steeply increase and then level off.
- The rise is caused by discharge of ion and leveling off is due to concentration polarization by diffusion. The height of rise in current depend on the concentration of ion in the solution.

- After one ion type in the solution is completely discharged, further ion will discharge as its decomposition potential is reached.
- Hence current-voltage curve of a solution will show a no. of such waves, each wave corresponding to the discharge of each type of ion. Height of wave represents the quantity of ion present.
- Useful for analysis of substances that are subjected to electrolytic oxidation and reduction
- Analysis of all metals, non ferrous alloys, steels, Mg alloys, Zn alloys & ores.

Chromatograph



Gas chromatography

- Continuous measurement of amount of each constituent in a complex vapor or gas mixture.
- Sample gas is mixed with a carrier gas (H2, air, N2 or He) and passed through a packed chromatographic column, which is made of metal tubing and filled with an adsorbent such as activated Al, silica gel or activated carbon and operated at a suitable temperature.
- The rates at which individual component move through the column depend on their respective affinities for the column material. Hence different components emerge from the column in a sequence which depend on the relative affinity of the components for the particular column packing.

- A GC neither identifies nor measures components directly. For this purpose employs a device which detects each component as it emerges out of the column and send signal to the recording device.
- The identification of a component is made by measuring the time required for a component to move through a column operating under specified conditions; and a quantitative determination is made by measuring peak area or height.
- Two basic sections of chromatograph – analysis section and control section

Analysis Section

- Chromatographic valves – Sample valve to introduce a fixed volume sample into the carrier stream and column valve to switch segments of the column out of and into the carrier systems employed. Column switching valves to flush out unwanted components from precut column. Types of valves used – slide, spool, rotary and flexible diaphragm. Capable of remotely controlled, pneumatically operated, leak proof, high quality
- Columns – Consists of small diameter (3 to 6 mm) coiled tubing packed with a bed of material which offers varying degree of resistance to the stream components.

The separation efficiency of a column is affected by following factors – bed particle size, liquid substrate, column length, column temperature.

- Detectors : Thermal conductivity cells using thermistors as sensing device. A metered quantity of sample gas is passed through the measuring chamber and carrier gas is passed through the reference chamber. The output of the cell is amplified and applied to the recording instrument (computer) to obtain chart showing different peaks at different times.
- Analyser housing is designed for hazardous areas, provided with a temperature controlled enclosure, capable of absorbing physical shocks.

Control Section

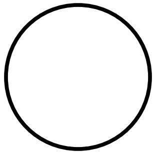
- Programmer – to amplify and transmit signal to recorder/ controller, commands the sample , column valves and stream switching system, selects chromatogram mode, calibration mode and automatic analysis. Components – timer, power supply, automatic zero mechanism, attenuation pots.

P & I diagrams

- Uses of P&IDs
- Develop Operational methodology
- Develop Safety Philosophy and Design
- Develop Control Philosophy
- Basis for Control Programming
- Communication Document for Project
- Serves as Design Basis for:
- Equipment Design ,Piping Design, Estimating Purchasing
- Used to evaluate construction progress
- Training basis for Operational Personnel

Development of P & I diagrams

Instrumentation Symbology

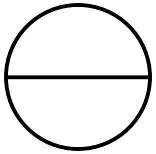


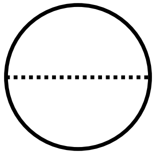
Instruments that are field mounted

-Instruments that are mounted on process plant (i.e sensor that mounted on pipeline or process equipments.

Field mounted on pipeline

Instrumentation Symbology

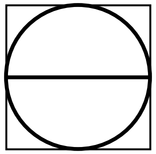




Instruments that are board mounted (invisible).

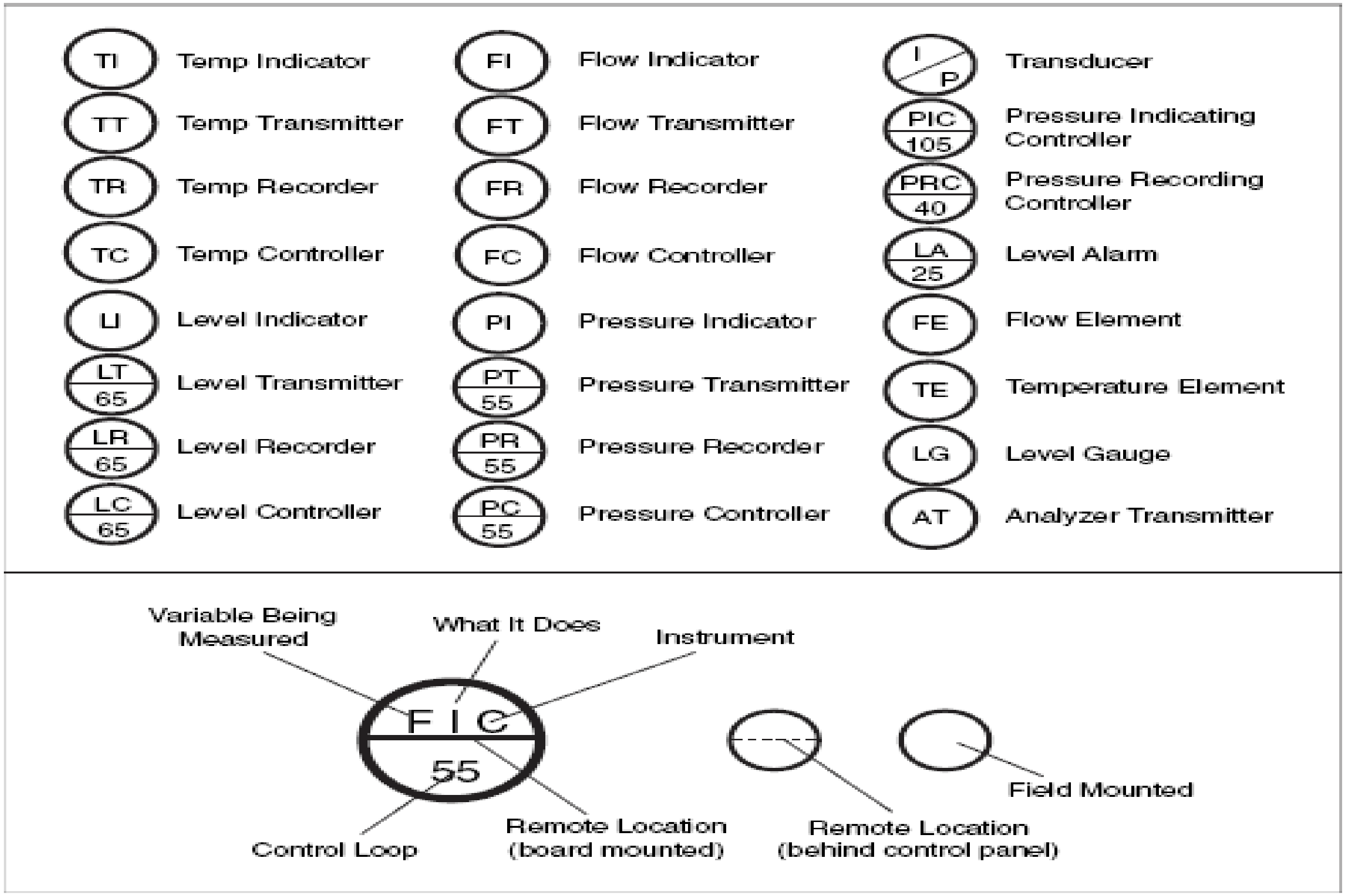
-Instruments that are mounted behind a control panel board.

Instrumentation Symbology



Instruments that are functioned in Distributed Control System (DCS)

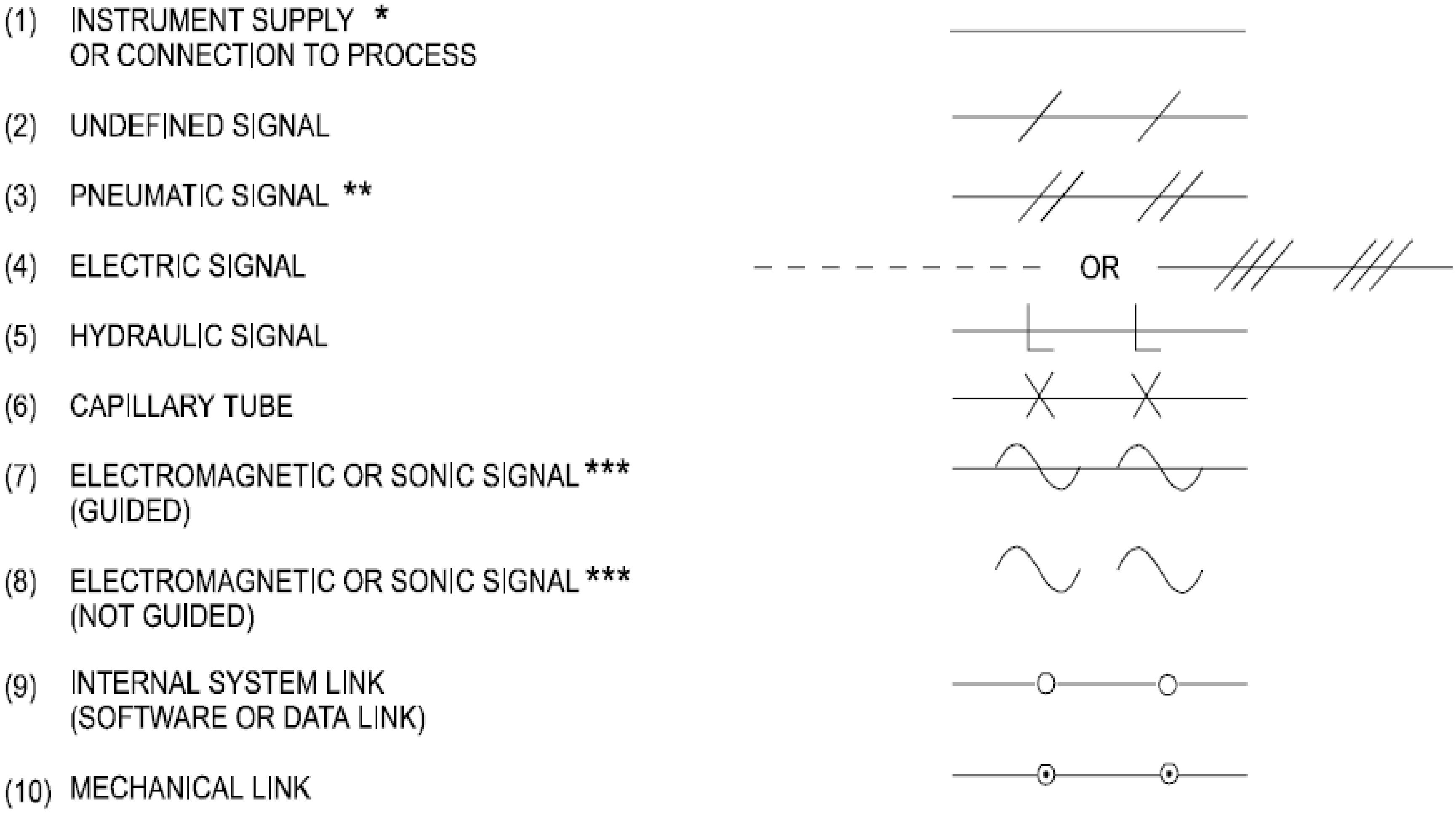
- A **distributed control system** (DCS) refers to a control system usually of a manufacturing system, process or any kind of dynamic system, in which the controller elements are not central in location (like the brain) but are distributed throughout the system with each component sub-system controlled by one or more controllers. The entire system of controllers is connected by networks for communication and monitoring.


TI Temp Indicator
TT Temp Transmitter
TR Temp Recorder
TC Temp Controller
LI Level Indicator
LT 65 Level Transmitter
LR 65 Level Recorder
LC 65 Level Controller
FI Flow Indicator
FT Flow Transmitter
FR Flow Recorder
FC Flow Controller
PI Pressure Indicator
PT 55 Pressure Transmitter
PR 55 Pressure Recorder
PC 55 Pressure Controller
I P Transducer
PIC 105 Pressure Indicating Controller
PRC 40 Pressure Recording Controller
LA 25 Level Alarm
FE Flow Element
TE Temperature Element
LG Level Gauge
AT Analyzer Transmitter
Variable Being Measured
What It Does
Instrument
F I C
55
Control Loop
Remote Location (board mounted)
Remote Location (behind control panel)
Field Mounted

FC	Flow Controller
FE	Flow Element
FI	Flow Indicator
FT	Flow Transmitter
FS	Flow Switch
FIC	Flow Indicating Controller
FCV	Flow Control Valve
FRC	Flow Recording Controller
PC	Pressure Controller
PG	Pressure Gauge
PI	Pressure Indicator
PR	Pressure Recorder
PS	Pressure Switch
PIC	Pressure Indicating Controller
PCV	Pressure Control Valve
PRC	Pressure Recording Controller
PDI	Pressure Differential Indicator
PDR	Pressure Differential Recorder
PDS	Pressure Differential Switch
PDT	Pressure Differential Transmitter

PT	Pressure Transmitter
PTD	Pressure Transducer
LC	Level Controller
LG	Level Gauge
LR	Level Recorder
LT	Level Transmitter
LS	Level Switch
LIC	Level Indicating Controller
LCV	Level Control Valve
LRC	Level Recording Controller
TE	Temperature Element
TI	Temperature Indicator
TR	Temperature Recorder
TS	Temperature Switch
TC	Temperature Controller
TT	Temperature Transmitter

Signal Lines Symbology



(1) INSTRUMENT SUPPLY *
OR CONNECTION TO PROCESS

(2) UNDEFINED SIGNAL

(3) PNEUMATIC SIGNAL **

(4) ELECTRIC SIGNAL

(5) HYDRAULIC SIGNAL

(6) CAPILLARY TUBE

(7) ELECTROMAGNETIC OR SONIC SIGNAL ***
(GUIDED)

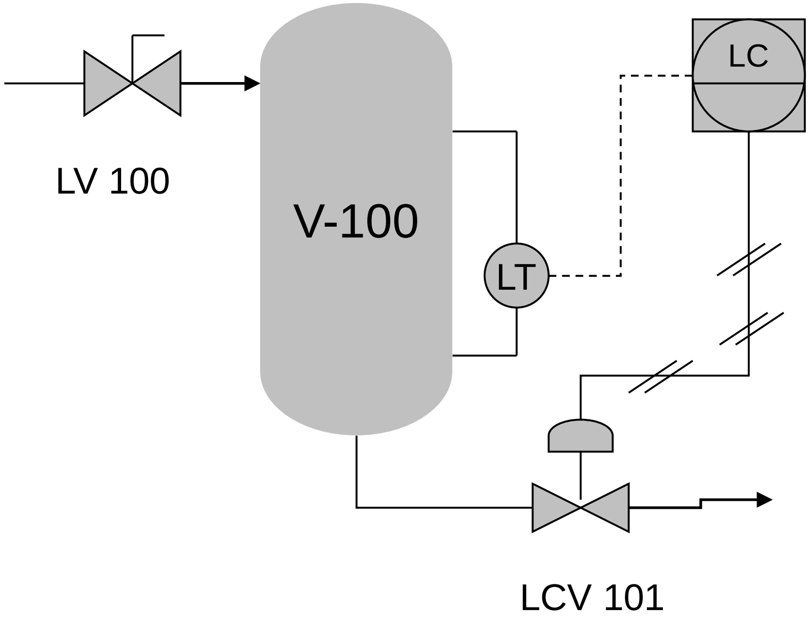
(8) ELECTROMAGNETIC OR SONIC SIGNAL ***
(NOT GUIDED)

(9) INTERNAL SYSTEM LINK
(SOFTWARE OR DATA LINK)

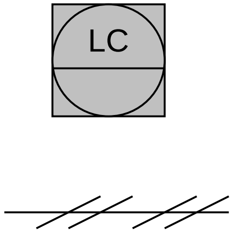
(10) MECHANICAL LINK

- Principle of P&ID

Example 1

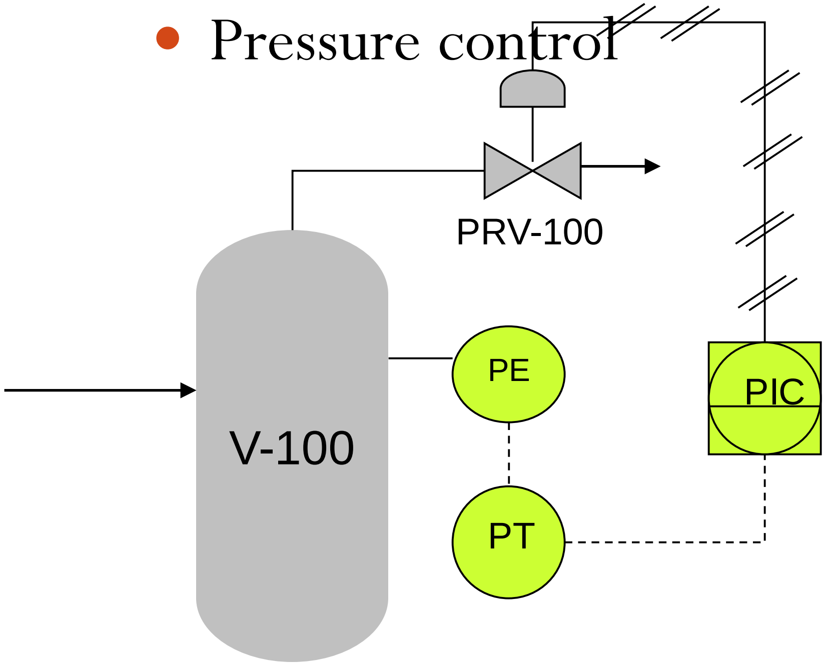


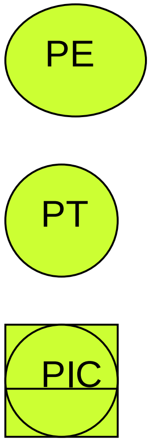
With using these following symbols;



Complete control loop for LCV 101

- Pressure control





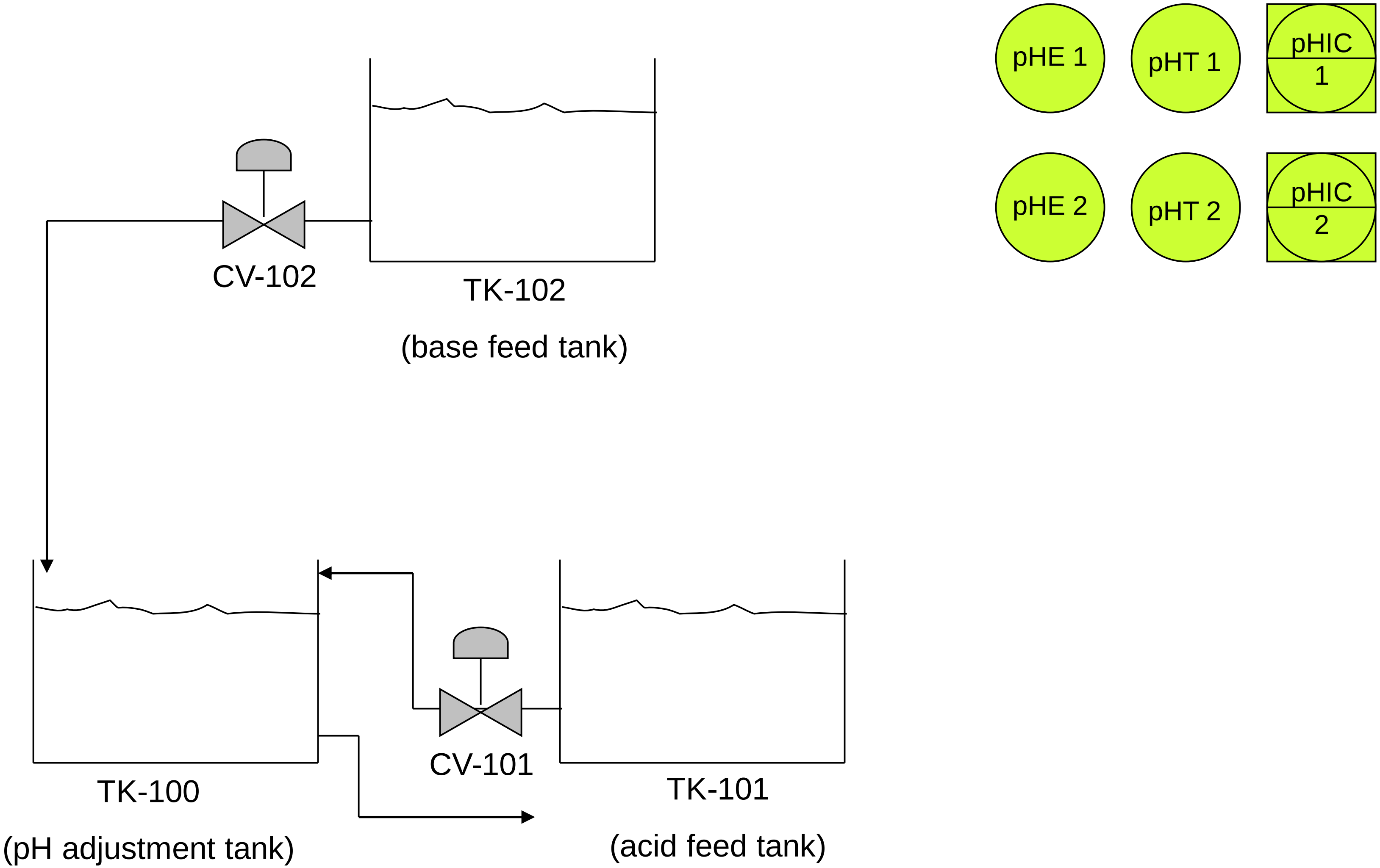
Where PE is locally mounted on V-100

Where PT is locally mounted

Where PIC is function in DCS

pH control P & ID

- The diagram shows pH adjustment; part of waste water treatment process. With using symbols given, draw control loop where the process need is:
- The process shall maintained at pH 6. When the process liquid states below pH 6, CV-102 will be opened to dosing NaOH to the tank TK-100. When the process liquid states above pH 6, CV-101 will be operated to dosing HCl.



pHE 1

pHT 1

pHIC
1

pHE 2

pHT 2

pHIC
2

CV-102

TK-102

(base feed tank)

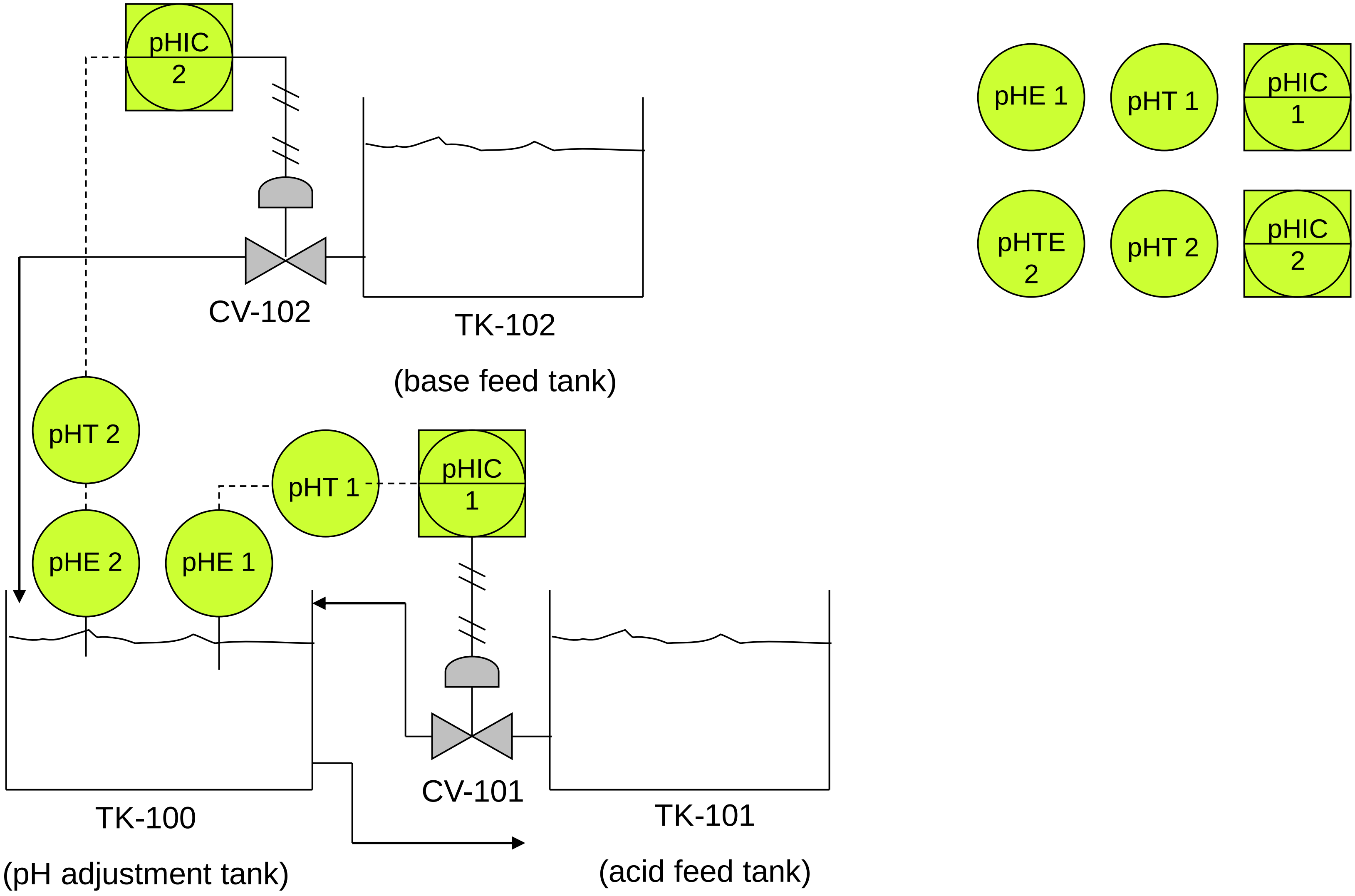
CV-101

TK-100

(pH adjustment tank)

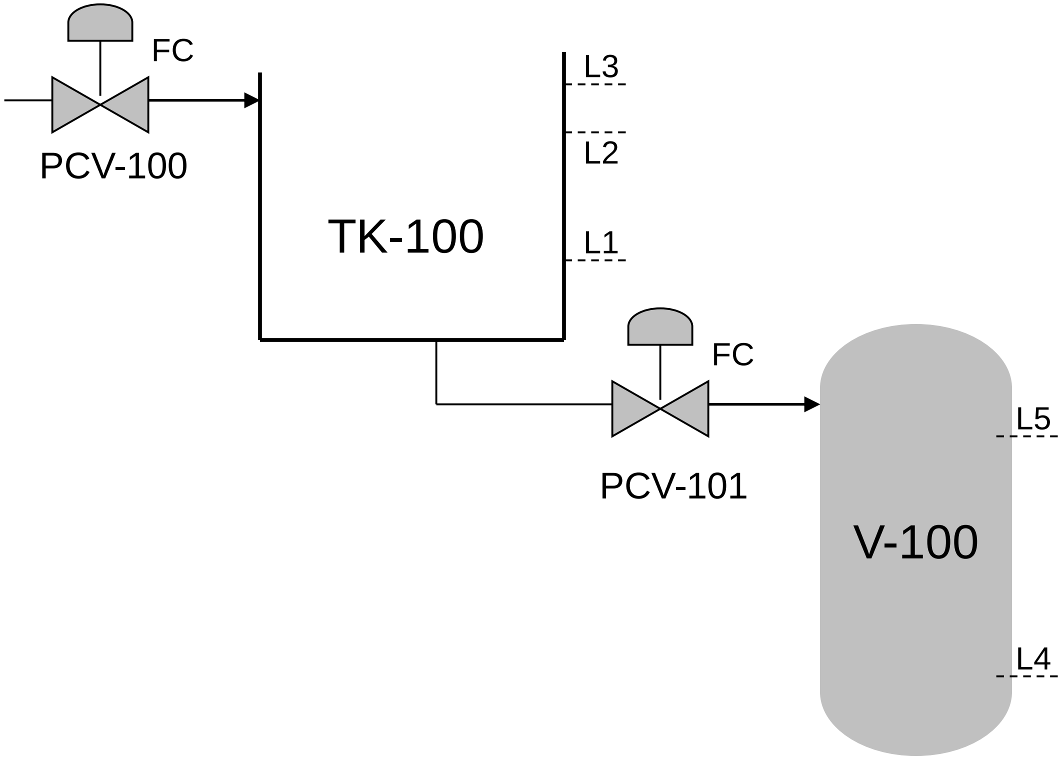
TK-101

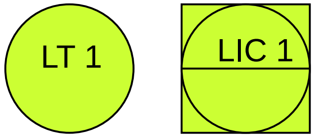
(acid feed tank)


pHIC
2
CV-102
TK-102
(base feed tank)
pHT 2
pHT 1
pHIC
1
pHE 2
pHE 1
CV-101
TK-100
(pH adjustment tank)
TK-101
(acid feed tank)
pHE 1
pHT 1
pHIC
1
pHTE
2
pHT 2
pHIC
2

Level control

Exercise 2

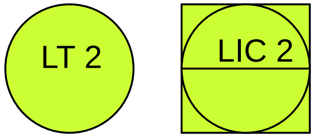




Where LT 1 and LIC 1 to control PCV-100 (failure close);

PCV-100 close when level reached L 3

PCV-100 open when level below L3

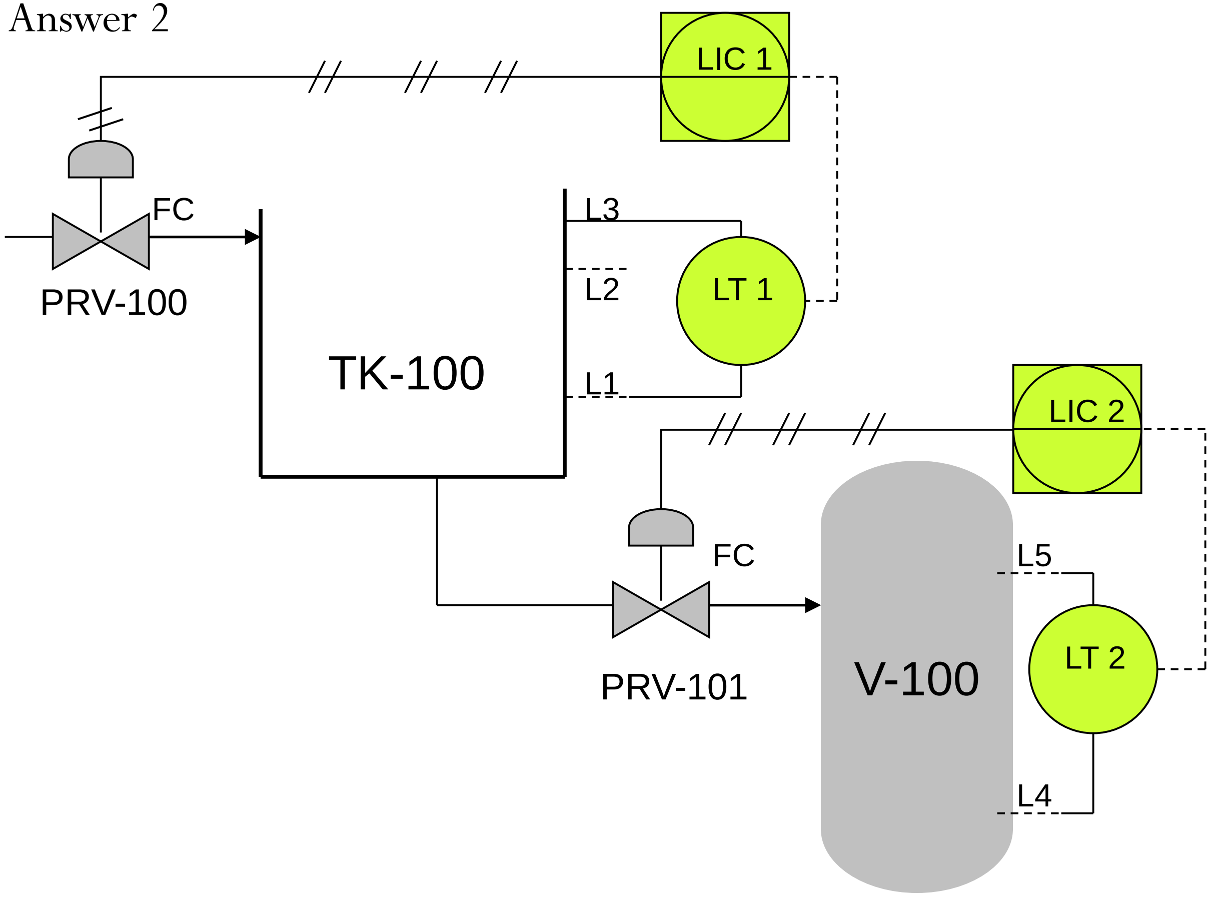


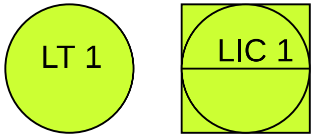
Where LT 2 and LIC 2 to control PCV-101 (failure close);

PCV-101 close when level reached L5

PCV-101 open when level below L5

Answer 2

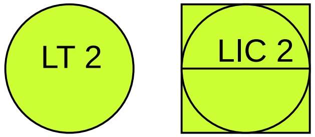




Where LT 1 and LIC 1 to control PRV-100 (failure close);

PRV-100 close when level reached L 3

PRV-100 open when level below L3



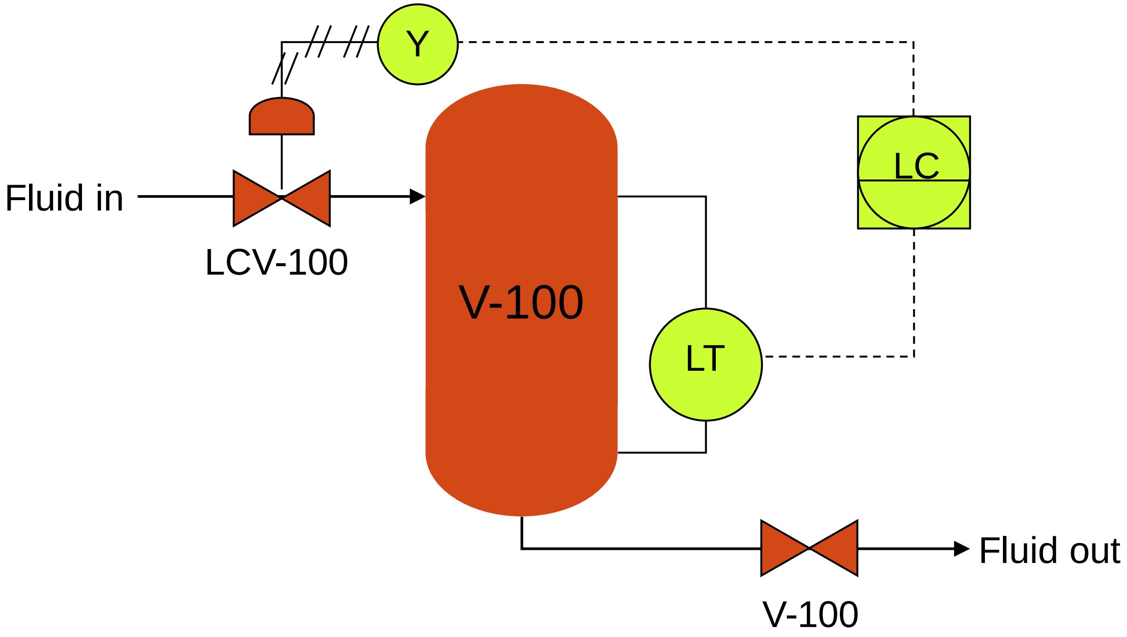
Where LT 1 and LIC 1 to control PRV-101 (failure close);

PRV-101 close when level reached L5

PRV-101 open when level below L5

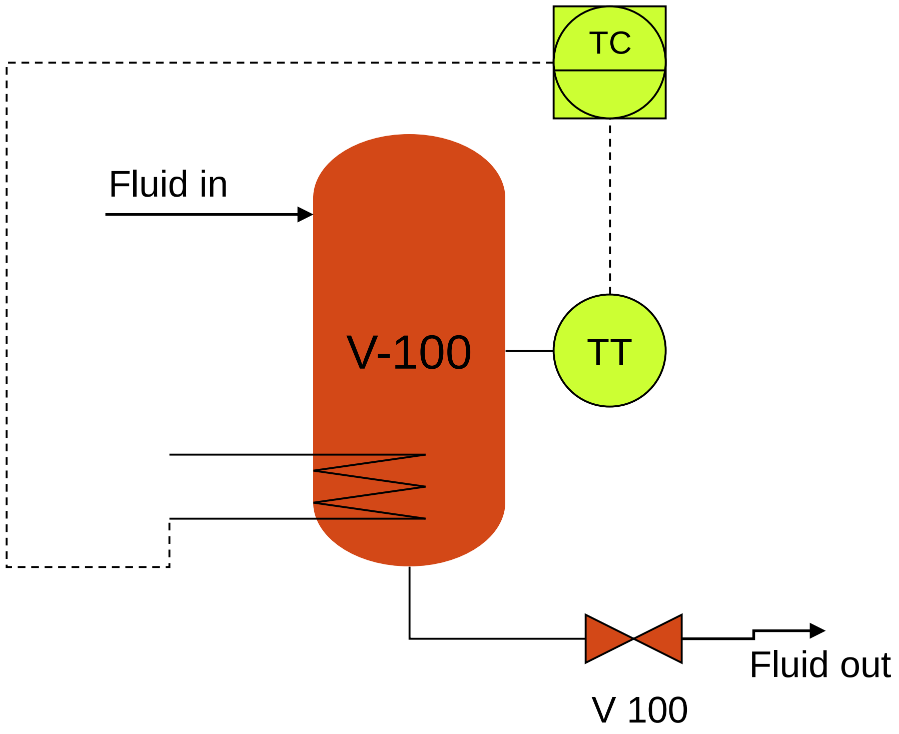
Feed back control

- One of the simplest process control schemes.
- A feedback loop measures a process variable and sends the measurement to a controller for comparison to set point. If the process variable is not at set point, control action is taken to return the process variable to set point.
- The advantage of this control scheme is that it is simple using single transmitter.
- This control scheme does not take into consideration any of the other variables in the process.


Y
LC
Fluid in
LCV-100
V-100
LT
Fluid out
V-100

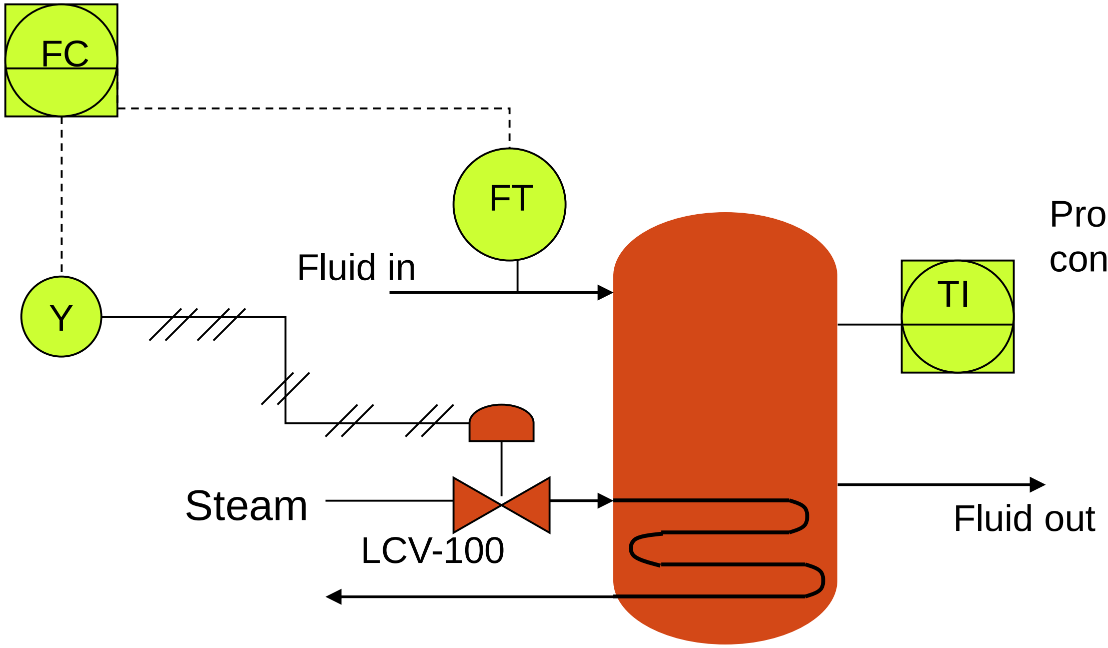
Example 1

- Figure below shows the liquid vessel for boiler system. This system has to maximum desired temperature of 120 degree Celcius (L2) where the heater will be cut off when the temperature reached desired temperature. Draw feedback control loop for the system.



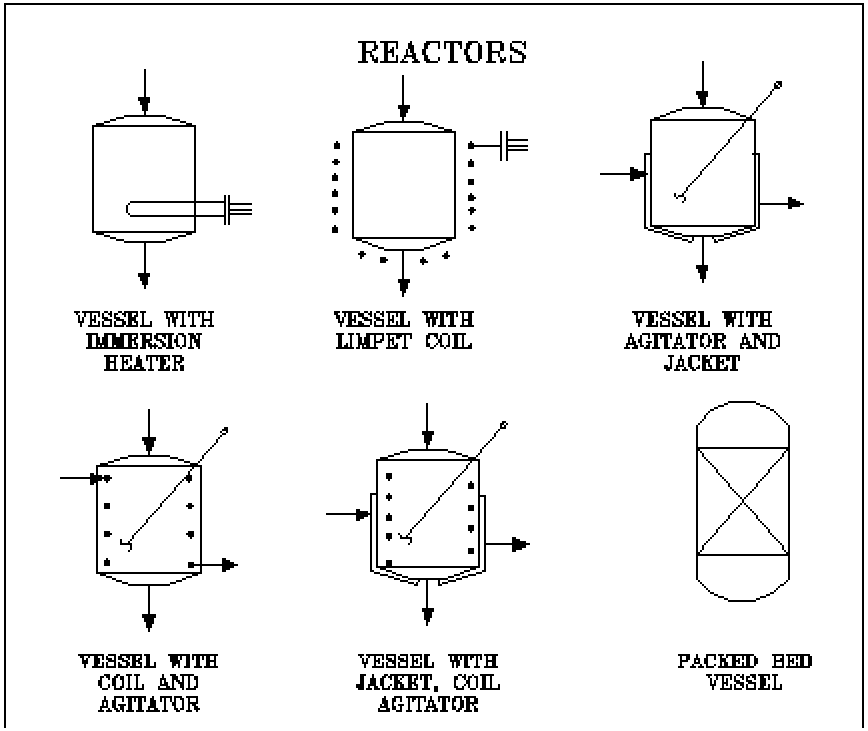
Feed forward controller

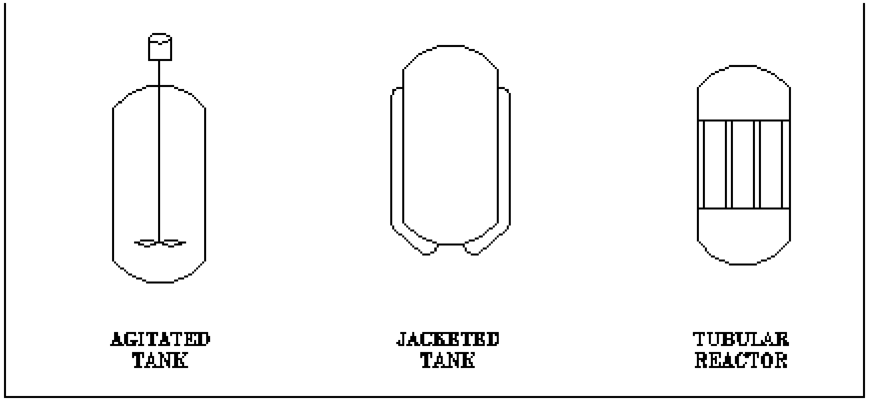
- Feedforward loop is a control system that anticipates load disturbances and controls them before they can impact the process variable.
- For feedforward control to work, the user must have a mathematical understanding of how the manipulated variables will impact the process variable.
- An advantage of feedforward control is that error is prevented, rather than corrected.
- However, it is difficult to account for all possible load disturbances in a system through feedforward control.
- In general, feedforward system should be used in case where the controlled variable has the potential of being a major load disturbance on the process variable ultimately being controlled.



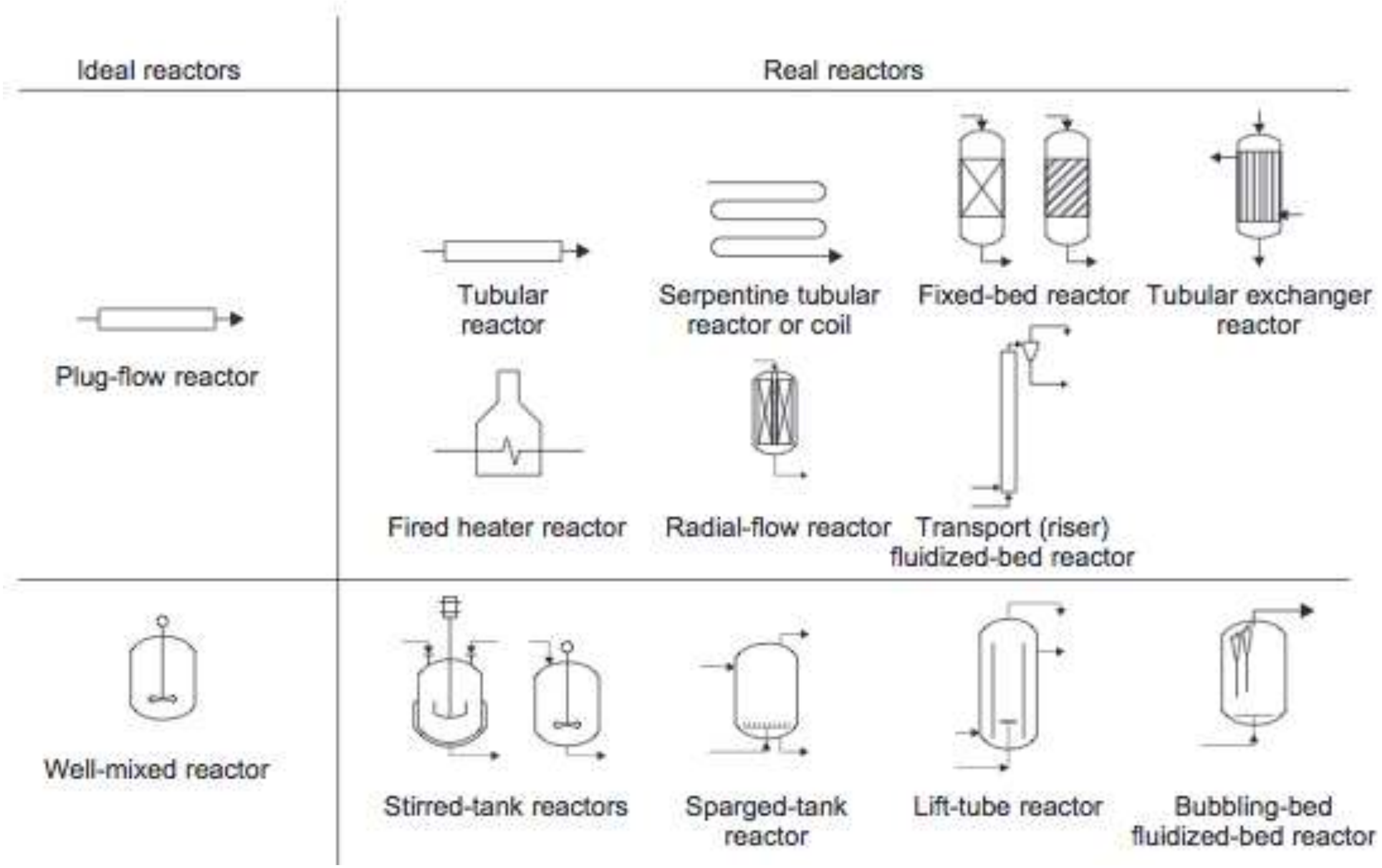
Process variable need to be controlled = Temperature

P & I diagrams for reactors

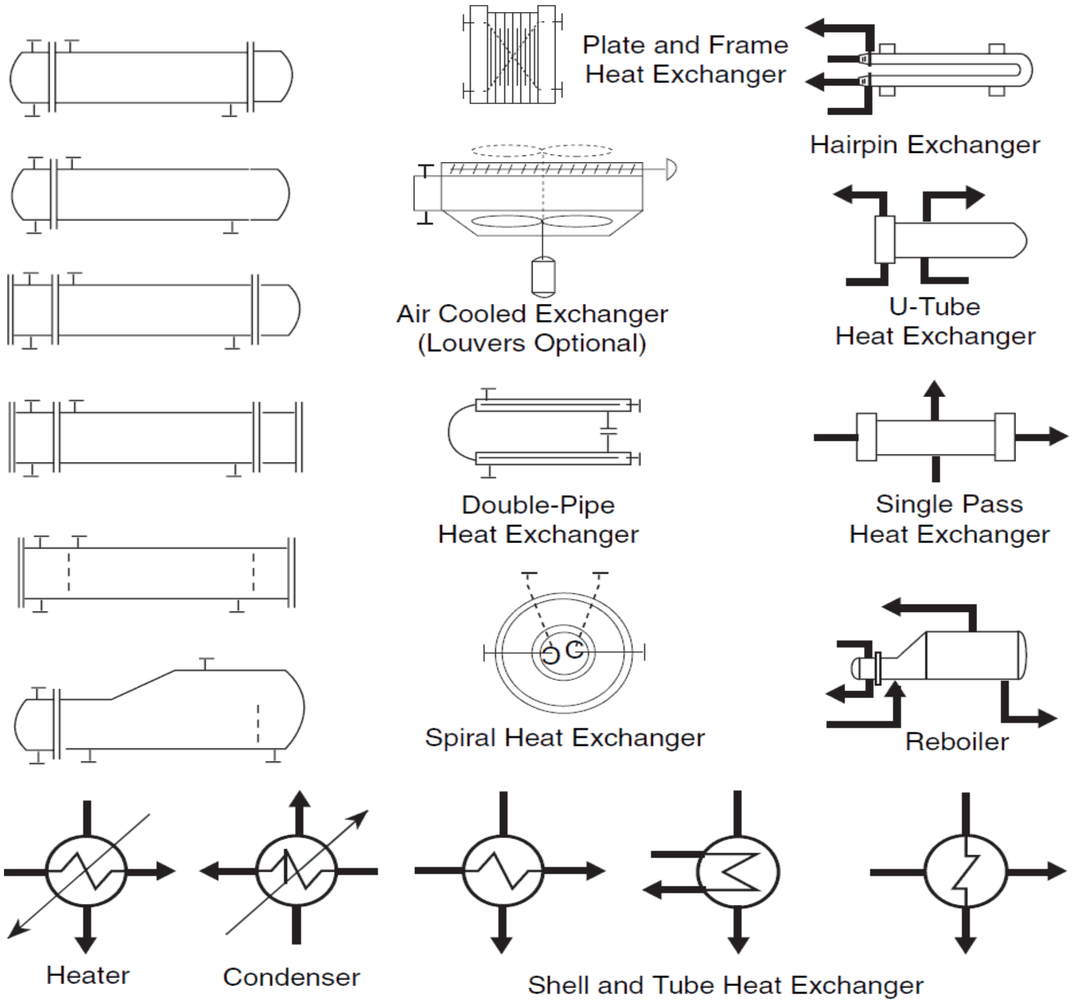

REACTORS
VESSEL WITH IMMERSION HEATER
VESSEL WITH LIMPET COIL
VESSEL WITH AGITATOR AND JACKET
VESSEL WITH COIL AND AGITATOR
VESSEL WITH JACKET, COIL AGITATOR
PACKED BED VESSEL



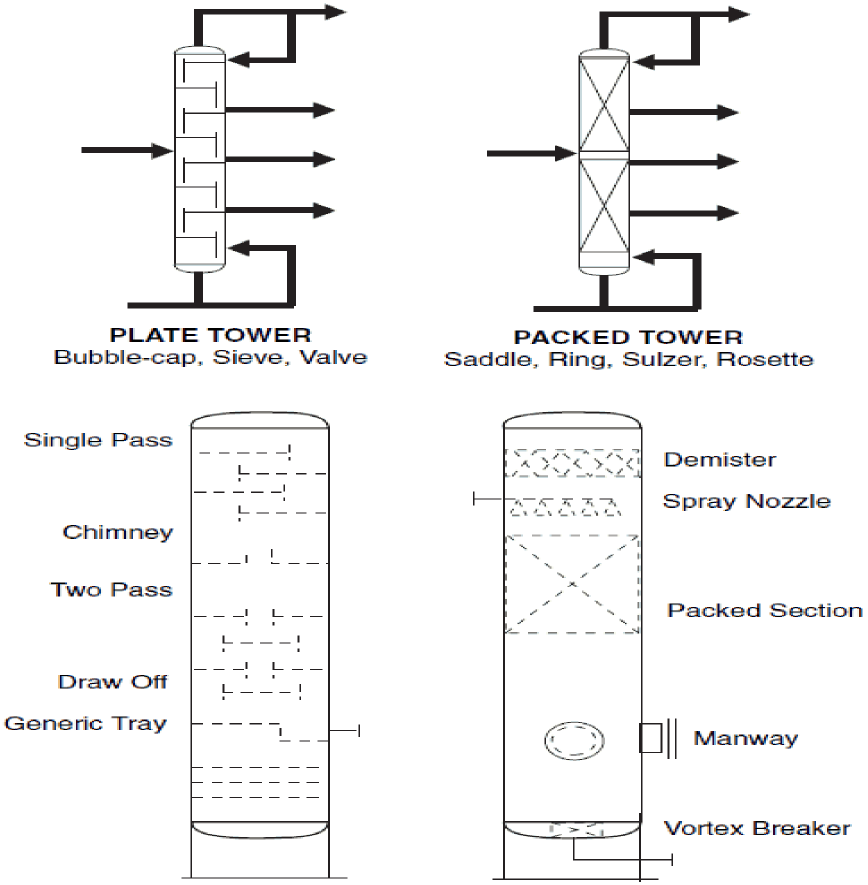
P & I diagrams for reactors



P & I diagrams -Heat exchangers



P & I diagrams- Distillation column



Thank You